

Interoffice Memorandum

To: Gordon MacLeod

Date: April 24, 1997

From: Kraig K. Knapp
Remediation Projects,

No.: E110-DC-97-0008

Subject: Bikini Water Sample TPH Results
Project No. 04002

This memo is in response to your request to examine analytical results for TPH analyses on some water samples from Bikini Island.

After talking with you on the telephone on April 21, and looking over the TPH sample results, the following points are important:

- The five samples were from very different sources.
- No quality assurance/quality control (QA/QC) samples, such as trip blanks or matrix spikes, are listed on the Chain of Custody form.
- Samples were collected and shipped in Nalgene® bottles.
- As noted in the laboratory report, chromatograms of the samples did not match that of diesel.
- Samples were injected about every 46 minutes. The diesel standard was injected about 2 hours and 15 minutes after the last water sample.

The most striking feature of the chromatograms is their nearly identical appearance. The "fingerprint" of these samples and their overall intensity are almost perfect overlays. Considering that these water samples came from such different sources, it is not possible for them to be contaminated by the same analytes and at the same concentration. This indicates contamination occurred during or after sample collection. The most likely culprit is the plastic bottles used to hold and ship the water samples. Nearly all plastics contain additives which modify their properties. Most plastics are rather brittle unless a plasticizer is added. The usual chemicals used are called phthalates. Another common additive is an antioxidant, such as BHA or BHT, which slows deterioration. Nalgene® bottles have large quantities of both, as well as left over organic materials used in its manufacture. Another piece of evidence is that the concentration of these unknown compounds does not change on subsequent injections. If the gas chromatograph had been contaminated, their concentration would drop on each subsequent injection. Their identical concentrations followed later by the diesel standard, which shows none of the contaminant peaks, clearly shows the samples were contaminated, and not the gas chromatograph.

It is unlikely that the samples you shipped to the second lab will have different results. Not only can the Nalgene® bottles leach contaminants into the water, but it can absorb hydrocarbons out of the water. These results would fail any certification. Resampling is strongly recommended.

The level of QA/QC depends strongly on the end use of the data. If this is to do a general check of the water because of a vague complaint, then it can be minimal. If there is any chance this data might be used in legal proceedings, or if it might be used for compliance determinations, then more rigorous procedures are required. From my own experience, data originally collected for one purpose has often been used for other reasons in order to lower costs, or simply because no other data are available.

Recommendations:

At a minimum, one liter of water has to be collected for each sample. Specially cleaned one liter glass bottles with teflon[®] lined caps and certificates of analysis need to be used. The laboratory performing the analysis may provide these bottles as part of the service, or you can purchase them. Our group (Remediation Projects) buys bottles from Eagle Pitcher (800-331-7425).

The Chain of Custody showed samples were extracted only three days after collection. This was excellent, since up to 14 days are allowed. The samples arrived at the lab at 25°C. Shipping protocol requires samples arrive at 4±2°C. We ship all samples in large ice chests, packing large quantities of ice around the samples.

TPH analysis normally does not use a field blank¹. Because of the long travel distance and isolated nature of your situation, I would recommend at least a trip blank². If a sample collection device is used to put water into the sample bottles, then you should also take an equipment blank³.

The laboratory included a sheet showing results from a laboratory control standard, LCS, and a matrix spike/matrix spike duplicate⁴ (MS/MSD). Chromatograms were not included and it was also not apparent what water was used to prepare the MS/MSD⁵. Normally one of the sample sources would be chosen at random and three samples collected. Each of these samples are put on a separate line in the Chain of Custody form. The second and third samples would have the same sample identification with MS and MSD added, respectively (e.g. #6-Kitchen [MS], then #7-Kitchen [MSD]). This is recommended when resampling.

The other QA/QC recommended is to take a duplicate. Two liters of water from one source is collected and treated as separate samples. Depending on requirements, the laboratory may or may not be told the sample is a duplicate. Duplicates are often run "blind" to the laboratory to check their precision under realistic conditions.

Remediation Projects has had a lot of experience in sampling for nearly every type of environmental matrix. I have been an auditor and worked a number of years in environmental labs. Please call me at 295-7186 if you need further assistance in these areas.

Notes:

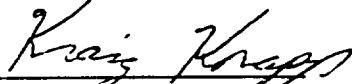
¹A Field Blank is an artificial sample designed to monitor the introduction of artifacts into the process. For aqueous samples, reagent water is used as a blank matrix. The blank is taken through the appropriate steps of the process. Field blanks are aliquots of analyte-free water brought to the field in sealed containers and transported back to the laboratory with the sample containers. Trip blanks and equipment blanks are two specific types of field blanks.

²Trip blanks are not opened in the field. They are a check on sample contamination originating from sample transport and shipping and from site conditions.

³Equipment blanks are opened in the field, and the contents are poured appropriately over or through the sample collection device, collected in a sample container, and returned to the laboratory as a sample. Equipment blanks are a check on sampling device cleanliness.

⁴Matrix Spike/Matrix Spike Duplicate Analysis uses predetermined quantities of stock solutions of certain analytes that are added to a sample matrix prior to sample extraction/digestion and analysis. Samples are split into duplicates, spiked, and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision. The concentration of the spike should be at the regulatory standard level or the estimated or actual method quantitation limit. When the concentration of the analyte in the sample is greater than 0.1 percent, no spike of the analyte is necessary.

⁵Matrix Spikes are actually laboratory QC samples, not field QC. However, field personnel may need to collect additional volumes of sample (replicates) for matrix spike and matrix spike duplicate analyses. The same volumes collected for the environmental sample must be collected for both the matrix spike and the matrix spike duplicate, (i.e., if two volatile organic analysis vials are collected for the environmental sample then two are collected for the matrix spike and two for the matrix spike duplicate).


Kraig K. Knapp

KKK:nms
Subject Code: ENV 19

cc: Correspondence Control, M/S NLV008
D. K. Cowser, M/S NLV082
K. A. Mobley, M/S NLV022
S. J. Nacht, M/S NTS306